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1. Cover Page:

Final Project Report

PI: Kimberly L. Turner
UC Santa Barbara
Mechanical Engineering Department
UC Santa Barbara
2355 Engineering Building II, Mailstop 5070
Santa Barbara, CA 93106-5070

Phone: 805-893-5106

Grant Number: FA9550-05-1-0045

2. Objectives: Same as originally stated

3. Status of Effort: Over the life of this grant, significant technical contributions have been made. When this grant commenced, we were at the very beginnings of building a hierarchical gecko-inspired adhesive. Since that point, we have developed hierarchical synthetic adhesive has been developed which utilizes milli/micro/nanofabrication to build active adhesive devices. In addition, in the final year of the grant, the adhesive was made actively reversible, and we were able (with additional funds from the ARMY UARC Institute for Collaborative Biotechnology) to realize the first bio-inspired REVERSIBLE adhesive, capable of sticking and unsticking with an integrated magnetic actuator.

Geckos, as well as many insects, have evolved a robust reversible adhesion mechanism, enabling them to traverse rough, smooth, vertical or inverted surfaces. In this final report, we present a synthetic reversible adhesive composed of flexible nickel paddles coated by aligned vertical polymeric nanorods. When subjected to a magnetic field, the nickel paddles undergo a reversible conformational change, greatly reducing the contact area, and decreasing adhesion by a factor of 40. In addition, the ratio of adhesion force to pre-load force achieved was 1.5, which is over an order of magnitude greater than any other research results to date on gecko-inspired synthetic adhesives. In the final year of the project, we were able to make this device fully reversible, capable of "unsticking" due to the application of the magnetic field. This represents a large step in the development of such systems for many applications. Such controllable adhesion may impact technologies ranging from ubiquitous latching systems to high-tech applications such as microrobotics.

Following the early results, we were able to utilize these results to obtain significant support from other branches of the department of Defense as well, broadening our goals.

4. Accomplishments/New Findings (over the life of the grant):

The mechanism of adhesion in the gecko has been of scientific interest since Aristotle (1). Since then scientific investigations have revealed much about the construction of the pad in the gecko's foot (2-6). Most recently there has been an intensifying scientific investigation into the fundamental physics of the adhesive, isolating van der Waals as the primary source of adhesion (7, 8), with additional evidence that humidity may also play an important role (9, 10). Van der Waals interactions produce weak and short-range forces, therefore the gecko must create a large amount of intimate surface contact to have enough adhesion to hang from a vertical or inverted surface. The gecko accomplishes this with a highly compliant pad structure, which allows it to conform to surfaces, without creating a large amount of elastic repulsive force (11). This ability to comply to a wide range of surfaces, from the curvature of a tree branch to micro- and nano-scale roughness of bark, is a result of a multi-scale compliant structure (12-15). The hierarchical structure consists of 200 nm wide, 5 nm thick spatulas at the ends of ~100 μm long, ~5 μm diameter setae (2, 3, 5, 16). The fine and thin spatulas conform to nanoscale roughness of a surface, enhancing the van der Waals forces and increasing adhesion through a contact splitting phenomenon (17). The setae provide the next level of surface compliance by bending to conform to micro- and millimeter scale roughness.

Without the compliance of the setal stalks, the spatulae would not come into contact with even the most moderately rough surface, greatly affecting the adhesive properties. There is evidence that the hierarchical structure may serve another purpose than enhancing adhesion – to reduce adhesion (7, 18, 19). As interesting as the gecko adhesion mechanism is, if the gecko were unable to release a surface, it would not be possible to take the next step. The nano/micro-scale components integrated into a hierarchical attachment/detachment structure allows the gecko to control adhesion at the nano-scale through macroscopic muscle movements (8, 11, 19).

Previously, a bio-inspired synthetic system enhancing adhesion utilizing a hierarchical structure was fabricated and tested (20). The system consisted of aligned vertical nanorods coating thin silicon dioxide platforms. The nanorods provided sufficient short-distance interactions to provide adhesion and the platform provided the bulk scale conformity necessary to adhere to rough or contoured surfaces. The combination of both structures provided increased adhesion over either isolated component. However, unlike the gecko, the system did not provide a mechanism for decreasing adhesion. This attribute is critical to any application of such a biomimetic system.

We have developed a new biomimetic system which provides a mechanism for decreasing adhesion using a magnetic field to actuate nickel cantilevers. The nickel beams, when placed in a magnetic field, reorient themselves so that the terminal pad of the structure, responsible for adhesion, rotates to face away from an adhering surface, Fig. 2. This conformational change effectively switches off the structure's ability to adhere by drastically reducing the available adhesive area.

Further development of reversible adhesive systems will lead to a new class of materials, able to stick and unstick controllably. These controllable adhesives may find applications ranging from everyday consumer products; such as a non-mechanical car door latching and sealing system; to improving manufacturing techniques with the ability to grip just about any surface; to high-tech niche applications, e.g. microrobotics. Just as this adhesive motif enables creatures such as beetles, spiders, and geckos to climb up and over objects, controlled adhesion will enable small scale robots to surmount obstacles of all sizes – allowing for the exploration of environments inhospitable or inaccessible to man, e.g. the surface of mars or the inside of a burning building.

Fabrication of the multi-scale structures required the integration of two different processing modalities. The nickel platform microstructures were photolithographically defined and etched using standard microfabrication reactive ion etching. The vertically aligned polymeric nanostructures were created through a stochastic growth method. Both methods employ batch fabrication techniques and are scalable to production quantities.

Released 150 nm thick and 130 μm long nickel structures, coated with aligned vertical arrays of stiff polymeric nanorods ~ 200 nm in diameter and ~ 3 μm tall, were fabricated using a combination of compatible massively parallel fabrication techniques. The fabrication process began by coating blank 4-inch (100) silicon wafers with a 1.4 μm thick layer of image reversal photoresist (AZ 5214). The negative image of the desired platforms was then transferred into the resist across the entire wafer using a Karl Suss MA6 contact aligner. After developing, a 150 nm thick nickel layer was electron beam evaporated onto the entire wafer. The photoresist was then removed, via an ultrasonic acetone bath, lifting off the excess nickel. The wafer was cleaned and dried and a 7 μm

layer of photoresist was spun onto the wafer surface (Shipley SPR 220-7). The positive pattern of the platforms was then transferred into the resist, aligned with the nickel platforms below. The resist and nickel pattern was transferred into the exposed silicon alternating between a highly reactive mostly isotropic SF_6 etch and a C_4F_8 passivation deposition (the Bosch process) effectively etching vertically into the silicon. After etching approximately 30 μm into the silicon, a sustained SF_6 etch was performed to undercut the nickel/photoresist platforms. The released platforms were then placed in oxygen plasma with an applied bias between wafer and plasma, creating ~ 200 nm diameter nanorods, orthogonally to the surface, with an aspect ratio of ~ 15 , Fig. 1.

The structures were characterized using a home-built adhesion test apparatus (Basalt II), Fig. 3 (21). The basic operating principle of the system is similar to an atomic force microscope, but implemented on a larger scale: the deflection of a glass spring is monitored, using laser interferometry, to determine the forces applied to the spring tip. This tip was a glass flat punch of 5 mm diameter. In order to ensure proper alignment between the tip and the sample, the tip was attached to the cantilever with high-strength glue while in intimate contact with the sample stage.

Test samples were placed on the micropositioning stage and moved to near contact with the spring tip. The tip was then lowered using a piezo electric actuator, and proper alignment was ensured through a horizontally oriented stereomicroscope. Actuation of the probe and data collection was performed using an automated National Instruments LabView™ program. Through calibration of the cantilever (spring constant, $k=137.1$ N/m) it was possible to determine the interaction forces between the flat punch tip and the test surface. Upon withdrawal from the surface, adhesion produced a characteristic pull-off event, evident in a negative dip of the force-displacement curve. The reversible adhesive was tested with and without Neodymium Iron Boron ($\text{Nd}_2\text{Fe}_{14}\text{B}$) rare earth metal magnet below the silicon chip.

While the gecko setae and spatulae are composed of β -keratin, here a combination of photoresist, silicon and nickel was used to create a 3-dimensional structure actuated through the application of a magnetic field. The photoresist ($E = 6.2 \pm 0.2$ GPa) is transformed into 200 nm diameter 3 μm tall nanorods, analogous to the β -keratin [$E = 1$ -15 GPa (22, 23)] spatulae of the gecko. These nanorods coat the thin nickel beams and act to enhance adhesion through contact splitting and nanoscale roughness conformation – thus acting as the active portion of the adhesive. The 150 nm thick nickel beams aid in surface conformation (just as the setae in the gecko) and as a deactivation mechanism for the adhesive. The stress mismatch between the photoresist and nickel causes the cantilevers to bend away from the surface. The upwards bend of these beams gives added compliance to a rough test surface by allowing individual cantilevers to bend and conform long before the test surface makes contact with the rigid adhesive substrate. In addition, the upwards bending of the beams isolates the active portion of the adhesive from the substrate.

With the active portion of the adhesive isolated, the properties of the adhesive could then be controlled by actuating the platforms. High-aspect-ratio ferromagnetic structures have been shown to rotate within a magnetic field to align their long axis with the magnetic field vector (24). When the structures were placed on top of a permanent magnet the paddles were observed to rotate about their long axis, Fig. 2. This rotation is attributed to the preferential alignment of the long axis of the width of the pad in the

magnetic field. In order to rotate the paddles in given direction, the stress inducing photoresist was offset on the paddles causing a slight pre-rotation, Fig. 4. The large rotation induced by the magnetic field causes the paddles to turn sideways, concealing the active portion of the adhesive from the test surface, Fig. 2.

Adhesion testing of the structures, without an applied magnetic field, produced unloading curves with a characteristic pull-off event shown in figure 4 (upper inset). The pull-off force was observed to vary with the maximum applied normal load (due to slight misalignments between the flat punch and the test surface) until a saturation adhesion strength of ~ 14 Pa was observed (obtained by dividing the adhesion force by the projected area of all pad surfaces), Fig. 4. It should be noted that this is a purely adhesive measurement testing in the normal pull-off direction, whereas reported values for the gecko test in the transverse frictional direction (25), making comparisons between the two systems tenuous.

Alignment issues, surface inconsistencies and unknown probe geometries have presented difficulties in quantification of this new class of bio-inspired non-pressure-sensitive-adhesives. One suggested metric is to simply divide the adhesion force by the maximum preload force, $\mu' = F_{\text{adhesion}}/F_{\text{preload}}$ (25). In this system the maximum μ' value was found to be 1.47 ± 0.4 , occurring at the minimum pre-load with an observable pull-off event (limited by the noise level of the instrumentation). This value offers a substantial increase from previous synthetic work with μ' values of 0.125 (20) and 0.06 (26), but still falls short of the gecko with $\mu' = 8$ to 16 (25).

In contrast to the adhesion seen in a rest state, the application of a magnetic field to the structures produced a catastrophic loss of adhesion, Fig. 4. The minimum negative force detected was 0.37 ± 0.28 Pa (compared with 14 Pa without a magnetic field). For no tests on the structures with an applied magnetic field was there an observable pull-off incident. This complete reduction in adhesion is attributed to the concealing of the nanorod-coated platforms from the test probe. Subjected to a magnetic field, the platforms rotate to align themselves with the magnetic field lines. The rotation leaves the edge of the platforms facing in the normal direction and the "sticky" face to the side. Thus when a surface approaches from the normal direction it only contacts the edges of the platforms. Since the edges of the platforms provide very little surface area, and have no nanorod coating, very little adhesion is produced – less than the noise in the instrumentation.

Additionally, a decrease in surface compliance was seen in the structures with an applied magnetic field. The twisting of the cantilevers increases the second moment of area of the structures, relative to the indenting tip, increasing the stiffness and consequently reducing the compliance of the system. Ultimately, the sideways turned paddles will contact the underlying substrate and statically block an adhering surface from contacting the support substrate – completely turning off adhesion.

In this paper, a novel approach has been presented for creating a synthetic analogue to the gecko adhesive system. The hierarchical system is composed of aligned vertical nanorods coating flexible micron scale cantilever paddles. The paddles, composed of nickel, rotate when subjected to a magnetic field. This rotation conceals the nanostructures on the paddle surface and greatly reduces the available surface area for adhesion. Testing of the system showed reversible adhesion behavior switching from a μ' value ($F_{\text{adhesion}}/F_{\text{preload}}$) of 1.47 ± 0.4 (largest reported value for a biomimetic system

to date (25)) to less than the noise level in the instrumentation. Thus an active hierarchical structure has been fabricated and demonstrated to display controlled and reversible adhesion. Further development of switchable adhesives will find applications ranging from everyday consumer products such as latching and fastening systems; to high-tech applications, such as enabling microrobotics to explore extraterrestrial surfaces or harsh climates otherwise not accessible to man.

In contrast to the adhesion seen in a rest state, the application of a magnetic field to the structures produced a catastrophic loss of adhesion (Fig. 3). The minimum negative force detected was (0.37 ± 0.28) Pa (compared with 14 Pa without a magnetic field). For no tests on the structures with an applied magnetic field was there an observable pull-off incident. This complete reduction in adhesion is attributed to the concealing of the nanorod-coated platforms from the test probe. Subjected to a magnetic field, the platforms rotate to align themselves with the magnetic field lines. The rotation leaves the edge of the platforms facing in the normal direction and the "sticky" face to the side. Thus, when a surface approaches from the normal direction it only contacts the edges of the platforms. Since the edges of the platforms provide very little surface area, and have no nanorod coating, very little adhesion is produced – less than the noise in the instrumentation. To test the reversible nature of the adhesive, the test probe was brought into contact with the adhesive surface monitoring the increase in load with time (Fig. 4). The probe was then retracted from the surface showing a decrease in normal load until the surface moved into the negative adhesive regime. At an arbitrary adhesive value the probe retraction was ceased, and either a Nd₂Fe₁₄B magnet was moved under the sample stage; or an electromagnet (doubling as a sample stage) was energized, inducing a magnetic field. In either case, repeatable catastrophic reduction in adhesion was observed (Fig. 4). Control experiments showed neither magnetic field had an effect on the glass cantilever and test probe. We presented a novel approach for creating a synthetic analogue to the gecko adhesive system. The hierarchical system is composed of aligned vertical nanorods coating flexible micrometer scale cantilever paddles. The paddles, composed of nickel, rotate when subjected to a magnetic field. This rotation conceals the nanostructures on the paddle surface and greatly reduces the available surface area for adhesion. Testing of the system showed reversible adhesion behavior switching from a $\mu 0$ value ($F_{\text{adhesion}}/F_{\text{preload}}$) of 1.4 ± 0.4 to less than the noise level in the instrumentation. Thus an active hierarchical structure has been fabricated and demonstrated to display controlled and reversible adhesion. The complete reversibility, which allows the switching of adhesion countless times, is the main advantage of the biomimetic approach presented here, especially over approaches based on shape-memory polymers. Further development of switchable adhesives, and improvement over the relatively low absolute adhesion strengths in this current system, will lead these adhesives to find applications ranging from everyday consumer products such as latching and fastening systems; to high-tech applications, such as enabling microrobotics to explore extraterrestrial surfaces or harsh climates otherwise not accessible to man.

These results are over an order of magnitude improved over any previously published van der Waals based adhesive devices. There are significant Air Force relevant applications which can be supported by this new technology. Satellite assembly/reassembly is one that personnel from the air force research laboratories have shown interest in. Further developments can focus more on this type of applications. The need for reversible adhesives which can function on a wide variety of surfaces is extremely broad. In terms of civilian technology, there is also significant applications from medical technology (bandages, adhesive surfaces for drug delivery, etc) to higher-quality post-it notes and reversible adhesives for everyday applications.

Figures

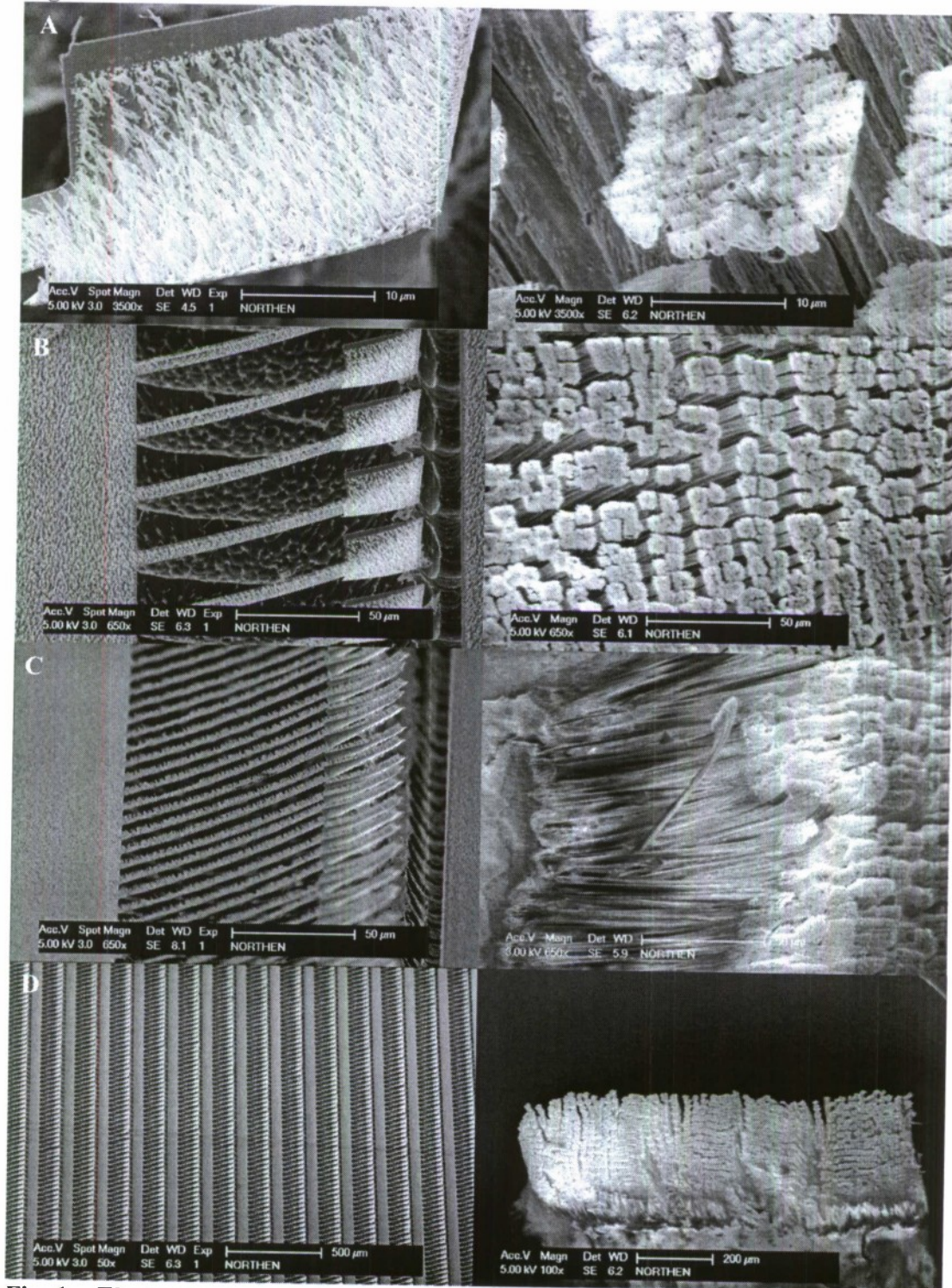


Fig. 1 – Electron micrographs of synthetic structures (left) and the analogous gecko structures (right), samples from a Tokay Gecko (*Gekko Gekko*). (A) Paddle surface coated with evenly spaced uncondensed aligned vertical polymer nanorods (left) and

the branched terminus of a seta into spatulae (right), same magnification and scale bar 10 μm . (B) Freestanding nickel cantilevers and paddles coated with nanorods (left) and an array of setae (right), same magnification and scale bar 50 μm . (C) Low angle view of cantilevers showing upwards bending of the structures relative to the solid substrate (left) and a profile view of curving setal stalks (right), same magnification and scale bar 50 μm . (D) Lower magnification view of a portion of the synthetic array (left) and the setal array (right), scale bars 500 μm (left) and 200 μm (right).

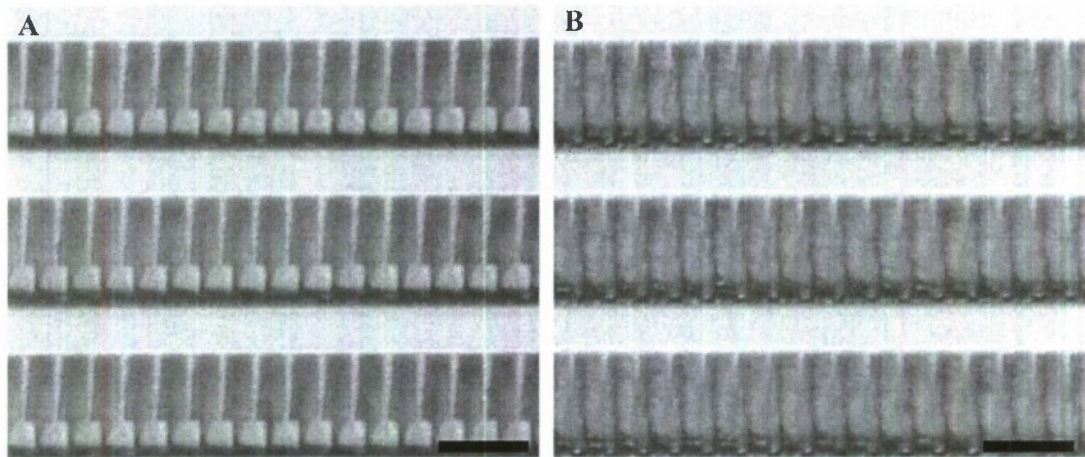


Fig. 2 - Stereomicrographs of the adhesive: (A) in the 'ON' state, no applied magnetic field, with the adhesive paddles facing vertically; and (B) in the 'OFF' state, with an applied magnetic field rotating the paddles sideways, concealing the adhesive faces. Scale bars, 100 μm .

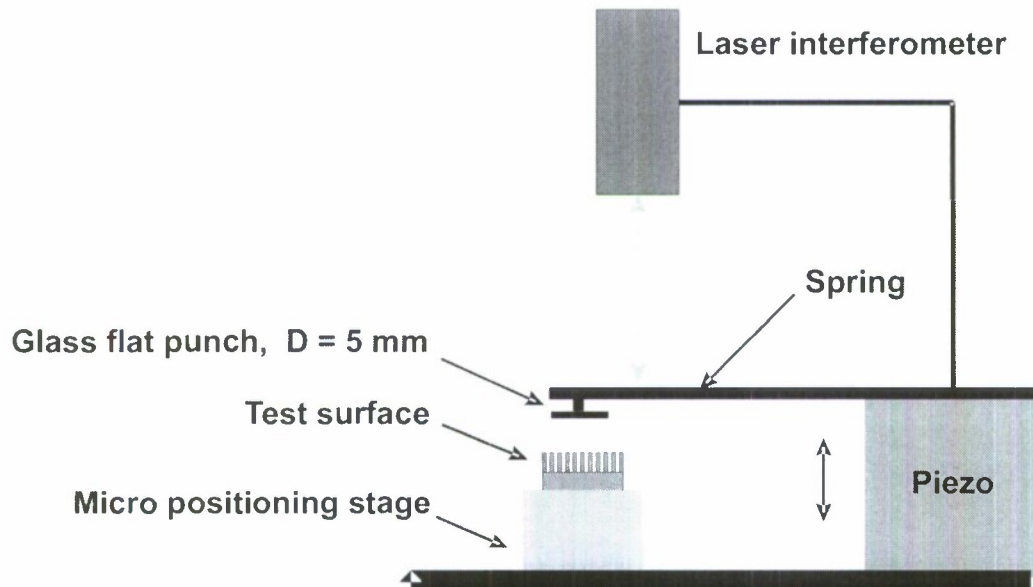


Fig. 3 – Schematic of the adhesion test apparatus. A laser interferometer monitors the deflection of a glass cantilever spring as a piezo actuator moves a 5 mm glass flat punch into and away from the test surface. The interaction forces are calculated by relating the stiffness and deflection of the cantilever upon contact with the surface.

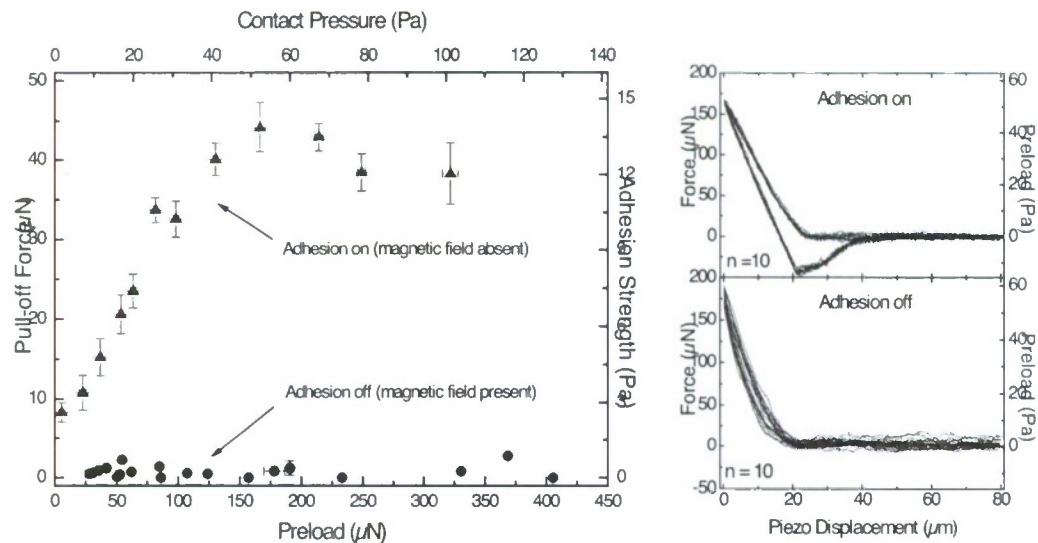


Fig. 4 - Adhesion results showing the on/off behavior of the structures without and with an applied magnetic field, respectively. The insets represent actual adhesion data, where in the 'ON' state distinctive pull-off events were observed (top) and in the 'OFF' state no pull-off events were observed (bottom). Strength values were obtained by dividing the interaction force by the contact area of the paddles. In the 'ON' state, the devices showed an initial increase in adhesion with preload force, characteristic of increased surface contact with applied load (likely a result of slight misalignment between the 5 mm flat punch and test surface). Error bars represent 10 data sets at a specified displacement with no emission of outliers.

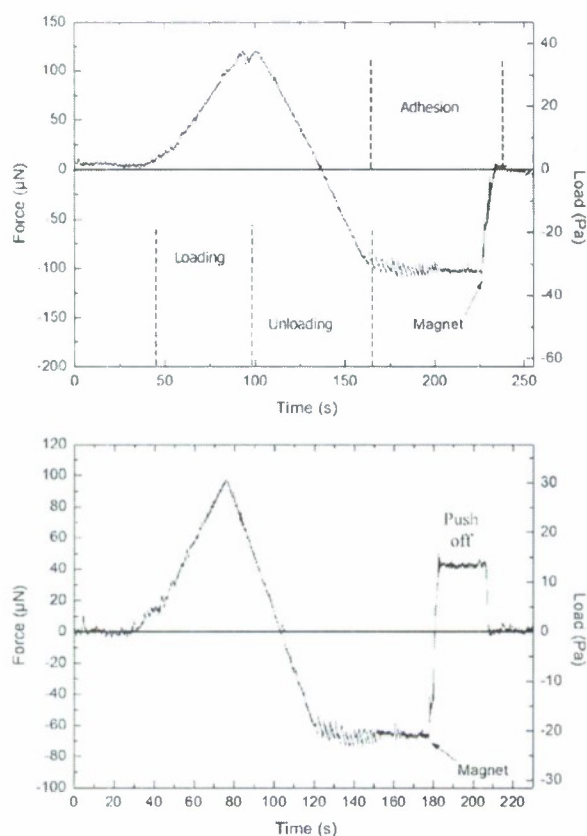


Figure 4. Adhesion results showing the release of the probe by the test surface. (Top) Plot showing a test probe contacting a test surface (indicated by the increase in positive normal force), then retracting from the surface (initiated at the maximum normal force), then moving into an adhesive regime (the normal load becoming negative), followed by the catastrophic loss of adhesion with the application of a magnetic field by moving permanent magnet under the sample holder (top) and by the application of an electromagnetic field (bottom).

Appendix: Experimental Procedures and Test Protocol.

Experimental

Micro/Nano-Fabrication: Released 150 nm thick and 130 mm long nickel structures, coated with aligned vertical arrays of stiff polymeric nanorods 200 nm in diameter and 3 mm tall, were fabricated using a combination of compatible massively parallel fabrication techniques. The fabrication process began by coating blank 4-inch (100) silicon wafers with a 1.4 mm thick layer of image reversal photoresist (AZ 5214). The negative image of the desired platforms was then transferred into the resist across the entire wafer using a Karl Suss MA6 contact aligner. After developing, a 150 nm thick nickel layer was electron beam evaporated onto the entire wafer. The photoresist was then removed, via an ultrasonic acetone bath, lifting off the excess nickel. The wafer was cleaned and dried and a 7 mm layer of photoresist was spun onto the wafer surface (Shipley SPR 220-7).

The positive pattern of the platforms was then transferred into the resist, aligned with the nickel platforms below. The resist and nickel pattern was transferred into the exposed silicon alternating between a highly reactive mostly isotropic SF₆ etch and a C₄F₈ passivation deposition (the Bosch process) effectively etching vertically into the silicon. After etching approximately 30 nm into the silicon, a sustained SF₆ etch was performed to undercut the nickel/photoresist platforms. The released platforms were then placed in oxygen plasma with an applied bias between wafer and plasma, creating 200 nm diameter nanorods, orthogonally to the surface, with an aspect ratio of 15 (Fig. 1).

Adhesion Testing: Test samples were placed on the micropositioning stage and moved to near contact with the spring tip. The tip was then lowered using a piezo electric actuator, and proper alignment was ensured through a horizontally oriented stereomicroscope. Actuation of the probe and data collection was performed using an automated National Instruments LabViewTM program.

Through calibration of the cantilever (spring constant, $k = 137.1 \text{ N/m}$) it was possible to determine the interaction forces between the flat punch tip and the test surface. Upon withdrawal from the surface, adhesion produced a characteristic pull-off event, evident in a negative dip of the force-displacement curve. The reversible adhesive was tested with and without Neodymium Iron Boron (Nd₂Fe₁₄B) rare earth metal magnet below the silicon chip or with an electromagnet (RS Components, Morfelden-Walldorf, Germany).

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5. Personnel Supported

Faculty: Dr. Kimberly Turner (PI)

Graduate Student Researchers: Michael Northen (finished Ph.D. in March 2006) and John Tamelier (Supported in 2006-2007)

6. Publications

Northen, M. T., Turner, K.L. (2005). "A batch fabricated biomimetic dry adhesive." *Nanotechnology* 16: 1159-1166.

Northen, M.T., Turner, K.L., Batch fabrication and characterization of nanostructures for enhanced adhesion. *Current Applied Physics*, 2005. 6(3): p. 379-383.

Northen, M.T., Turner, K.L., Meso-scale adhesion testing of integrated micro- and nano-scale structures. *Sensors & Actuators A-Physical*, 2005. 130-131: p. 583-587.

Michael T. Northen, Christian Greiner, Eduard Arzt and Kimberly L. Turner, A hierarchical gecko-inspired switchable adhesive, Solid-State Sensor, Actuator and Microsystems Workshop, Hilton Head Island, South Carolina, June 4-8, 2006

Michael T. Northen, Christian Greiner, Eduard Arzt and Kimberly L. Turner, A hierarchical gecko-inspired reversible adhesive, in press for Advanced Materials, 2008.

7. Interactions/Transitions:

a. Meetings/Conferences/Seminars

November 2005, Invited Seminar on Biologically Inspired adhesives given by PI Turner at Department of Mechanical Engineering, Brown University

November 2005, Attended/Participated in ASME IMECE, Orlando, FL

January 2006, Attended/Participated in MEMS 2006, Istanbul, Turkey

December 2005, Invited Seminar given by PI Turner at Mechanical Engineering, CalTech

May 2006 Invited Seminar/Collaboration meeting at Army Research Laboratory, ARL-SEDD, Adelphi, MD

June 2006 Oral Presentation at Hilton Head 2006 (Americas Workshop on Solid-State Sensors & Actuators).

May 2006 Spoke on biologically reversible adhesives at ARMY ICB Industry-Workshop, UCSB.

August 2006 Attended AFOSR Contractors meeting and presented year's research results

b. This work has attracted the interest of the ARMY for micro-robotic applications. Through the base AFOSR support, enough basic science results were achieved that the ARL became interested. Through additional seed funding (50K total), a collaboration has been started with ARL to try and develop additional technology more suited to the ARL applications. The AFOSR work is still focused on basic developments and applications most suited to AFOSR. However, were it not for this AFOSR grant, the ARL would have never known about this work.

c. The funding from AFOSR has been instrumental for technology transfer. A meeting occurred in May 2006 to discuss ARL transitioning for micro-robots. In addition, there are talks occurring with imt, inc., a MEMS foundry/design company to transition this technology as well. In 2007, a partnership was formed between ARL, IMT, Inc., and UCSB to further develop this work. A 6.2 project was funded by the ARMY/ICB to develop friction drives to enhance the switchable adhesion.

8. New Discoveries. Patent Disclosure/Provisional application filed March 2006, A biologically-inspired reversible adhesive. Inventors: Michael T. Northen and Kimberly L. Turner

9. Honors and Awards.

2005 Kimberly Turner received the UCSB Academic Senate Distinguished Teaching Award.

2006 Kimberly Turner chosen Technical Program Chair for Hilton Head 2008 (Americas workshop on Solid-State Sensors & Actuators)

2005-6 Kimberly Turner vice-chair of MEMS Division, ASME

2006 Kimberly Turner chosen for induction into Presidential Council of Alumnae, Michigan Technological University

2007 Kimberly Turner received Outstanding Young Alumni Award from Michigan Technological University